ILL RESEARCH PROPOSAL 88893

Printed: 15/02/2023

Title: **Resonant-enhancement signal and absorb-suppress noise for quantum-well resonator thin films**	Proposal Number (to be completed by ILL)

Proposer (to whom correspondence will be addressed)

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Peng PAN	**CHALMERS UNIVERSITY OF** Origovagen 6B GOTEBORG SWEDEN	±33638780185	pengp@chalmers.se

New neutron user: **No** New ILL user: **Yes**

Local contact contacted: **No**

Co-proposers

Name and first name	Laboratory	Country
KARLSSON Maths	**CHALMERS UNIV, GOTEBORG**	SWEDEN
LOHSTROH Wiebke	**MLZ, FRM II, TUM, GARCHING**	GERMANY
WOLFF Maximilian	**DEPT PHYS, UPPSALA UNIVERSITY**	SWEDEN

Local contact(s):

Suggested keyword number: **1-20**

This proposal is:

- [x] A new proposal
- [ ] A continuation proposal
- [ ] A resubmission

Main research area: **Physics**

Submitted to other facilities: **No** Societal indicators: **Fundamental Science**

Industry: **NOT related to industrial**

Unavailable dates:

Instruments

Requested instrument	Days
SUPERADAM	**4**

Requested starting time

- [ ] 1. Jan/Feb
- [ ] 2. Mar/Apr
- [ ] 3. May/Jun
- [x] 4. Jul/Aug
- [x] 5. Sep/Oct
- [x] 6. Nov/Dec

Comment:

Sample availability: **10*10*0.5 mm3**

Instruments' logical connection: **SUPERADAM**

Experimental details

Energy/Wavelength range:

Resolution in energy or wavelength:

Range of momentum transfer:

Resolution in momentum transfer:

Laboratory support facility

- [ ] Simulation support (C-lab)
- [ ] SAXS support through PSB
- [ ] Containment level 2 biology lab
- [ ] Chemistry Labs
- [ ] MSSL
- [ ] PSCM lab

Abstract

The measurement of hydrogen dynamics in thin films by neutron scattering is a significant technical challenge due to the drastically reduced sample volume. Neutron wavefield enhancement in a quantum-well resonator has recently developed a possible solution to this issue. To further validate the method and reduce the background scattering, we propose using triple resonators and high absorption substrate LiF, Fe/V superlattice grown on LiF and MgO. We will compare the results from PSD (neutron reflectivity) and incoherent scattering detectors, to check if the signal is higher enough for the dynamics experiment. If successful, we will, in a subsequent experiment, measure the inelastic/quasielastic scattering from such a resonator.

Publication

Influence of deuterium-induced volume changes on optical transmission in Fe/V (001) and Cr/V (001) superlattices, Droulias S.A., Granas O., Hartmann O., Komander K., Hjörvarsson B., Wolff M., Pálsson G.K., Physical Review B, (2022), 105, 195438-1-195438-6

Π-GISANS: Probing lateral structures with a fan shaped beam, Vorobiev A., Paracini N., Cárdenas M., Wolff M., Scientific Reports, (2021), 11, 17786-1-17786-8

Characterization of multiphase polymer systems by neutron scattering, Wolff M., null, (2011), null, null

Nuclear spin incoherent neutron scattering from quantum well resonators, Wolff M., Devishvili A., Dura J.A., Adlmann F.A., Kitchen B., Pálsson G.K., Palonen H., Maranville B.B., Majkrzak C.F., Toperverg B.P., Physical Review Letters, (2019), 123, 016101-1-016101-6

Grazing incidence neutron scattering for the study of solid-liquid interfaces, Wolff M., Frielinghaus H., Cárdenas M., Gonzalez J.F., Theis-Bröhl K., Softwedel O., von Klitzing R., Pilkington G.A., Rutland M.W., Dahint R., Gutfreund P., null, (2023), null, null

Neutron reflectivity for the investigation of coatings and functional layers, Wolff M., Gutfreund P., null, (2021), null, null

Time resolved polarised grazing incidence neutron scattering from composite materials, Wolff M., Saini A., Simonne D., Adlmann F., Nelson A., Polymers, (2019), 11, 445-1-445-12

Limitations of the kinematic approximation in neutron reflectivity measurements for the analysis of bilayers, Droulias S.A., Pálsson G. K., Hjörvarsson B., Wolff M., Journal of Applied Crystallography, (2018), 51, 1556-1563

Resonant enhancement of grazing incidence neutron scattering for the characterization of thin films, Perrichon A., Devishvili A., Komander K., Pálsson G.K., Vorobiev A., Laven R., Karlsson M., Wolff M., Physical Review B, (2021), 103, 235423-1-235423-12

Sample description

Substance/Formula: **F/V superlattice grown on MgO and LiF**

Mass (mg): **1000**

State: **thin film**

Size (mm3): **1000**

Surface area (mm2): **100**

Space group: **bcc**

Container:

Unit cell dimension:	a = **3**	b = **3**	c = **3**
T(k)= **300**	α = **90**	β = **90**	γ = **90**

Solvent SLD:

Particle SLD:

Sample environment equipment (supplied by ILL)

Environment equipment: **Neutron Spin Filter** **Polarisation Analysis, Guide Field at Sample**

Laser: **None**

Use of gas: **None**

Temperature range:

Pressure range:

Magnetic field strength:

Danger associated with ancillary equipment: ☐ **Yes** ☐ **Uncertain** ☑ **No**

Safety aspects completed at submission

No danger associated with this sample

Danger associated with the sample preparation: ☐ **Yes** ☐ **Uncertain** ☑ **No**

Danger associated with the sample handling: ☐ **Yes** ☐ **Uncertain** ☑ **No**

Type of waste to be processed after experiment: ☑ **Chemical** ☐ **Biological** ☐ **Nanoparticles** ☐ **Radioactive**

To be filled in by ILL

Sample environment code

NSF PA

Comments by Health Physics Officer and Safety Engineer

Resonant-enhancement signal and absorb-suppress noise for quantum-well resonator thin films

1. Scientific background

Surface science has advanced enormously in recent decades, especially after discovering 2D materials, it has become an increasingly crucial interdisciplinary field between physics, chemistry, crystallography, biology, and materials science. This is related to the growing interest in surfaces and interfaces among the scientific community and their importance for technological applications. A big challenge is understanding the structure and dynamics at surfaces and interfaces at the atomic scale [1,2]. Neutrons have properties that enable them to be used as a unique probe in material research. Their spin and low energy make them sensitive to magnetic induction in solids and suitable for investigating lattice vibrations and diffusive processes. Most important in this context for the present study is that neutrons interact with the nuclei, and neutron scattering has higher penetration through many materials compared to other techniques. In addition, the neutron is sensitive to different isotopes of the same element. Neutron spectroscopy [4] and neutron reflectometry (NR) [3] have been reported in studying hydrogen diffusion and concentration in bulk crystals. However, surface-sensitive neutron experiments are limited by the brilliance of current neutron sources. One way to overcome the low signal of neutron scattering from surfaces is using samples in form of quantum resonators. This concept has been proven for, e.g., metal hydrides [3] and are now developing as a powerful tool to investigate the structure, and potentially dynamics, in thin-film systems.

2. Previous studies

Recently, we have shown that thermodynamics of hydrogen in thin films of vanadium drastically changes [5] but only limited information on the chemical diffusivity is available. As a model system to study thin film diffusion effects, we chose $VH_{0.5}$, a system that has been thoroughly characterised in bulk by neutron scattering. However, the measurement of the hydrogen (H) dynamics in thin films is a big technical challenge due to the drastically reduced sample volume which results in the limited amount of scattered beam signal. Different strategies are developed to enhance the signal-to-noise ratio from H. Wave field enhancement was recently reported in refs. [1,3] as a powerful method. The actual sample Fe/V resonator deposited on a MgO substrate has been reported on Super ADAM at the ILL (see Fig. 2). The scattering signal recorded by two detectors (incoherent detector and PSD) [1], For this proposed experiment, we will install another incoherent scattering above the sample as shown in Fig. 1.

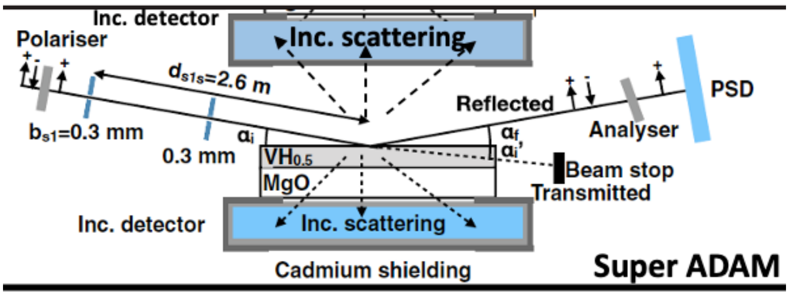

Figure 1. Schematic of the experimental setup at Super ADAM

3. Proposed experiment

The current proposal aims at improving the signal-to-noise ratio and demonstrating the feasibility of diffusion measurements in thin films, by neutron reflectometry (NR) on Super ADAM. The NR measurements will be performed on our new multi-stack Fe/V superlattice thin films deposited on LiF(001) substrate ($10 \times 10 \times 1$ mm^3), a new system for neutron wave-field enhancement quantum-well resonator. Comparing the NR results with the thin film on MgO(001) substrates, LiF and MgO have comparable crystal lattice constants of around 4 Å and both are bcc crystals. Nevertheless, the absorption cross section of LiF (Li: σ_{abs}=70.5 barn, F: σ_{abs}=0.0096 barn) is about 3 orders of magnitude larger than MgO (Mg: σ_{abs}=0.063 barn, O: σ_{abs}=0.00019 barn). The main background source of incoherent scattering from Mg [1] will be reduced. The quantum-well resonator consists of a material with different neutron scattering length densities (SLD), relatively high in two sides and low in the middle, thus forming a potential well for neutrons.

Crucially, we will take NR measurements and incoherent scattering on two films, differing in film thicknesses, sandwiched between Fe grown on LiF and one film grown on MgO. To evaluate whether such a quantum well resonator on LiF substrate could be used to reduce background scattering to study the dynamics of hydrogen.

Figure 2.b) shows the vanadium hydride thin films (triple resonator, 30 nm, 35 nm and 40 nm thick in one sample deposited on LiF and MgO substrates separately) prepared at Uppsala University, by established magnetron sputtering techniques [6]. This new sample will give resonances at larger Q. With previous 100 nm film we were only able to access the second resonance [1]. At the first resonance we should have more signal. Furthermore, it will also allow to accept more divergence and a larger spread of wavelengths. We believe LiF is an ideal substrate for the proposed experiments because of the high absorption cross section of Li (σ_{abs}=70.5 barn) and the low incoherent scattering cross section of F (σ_{inc}=0.0008 barn).

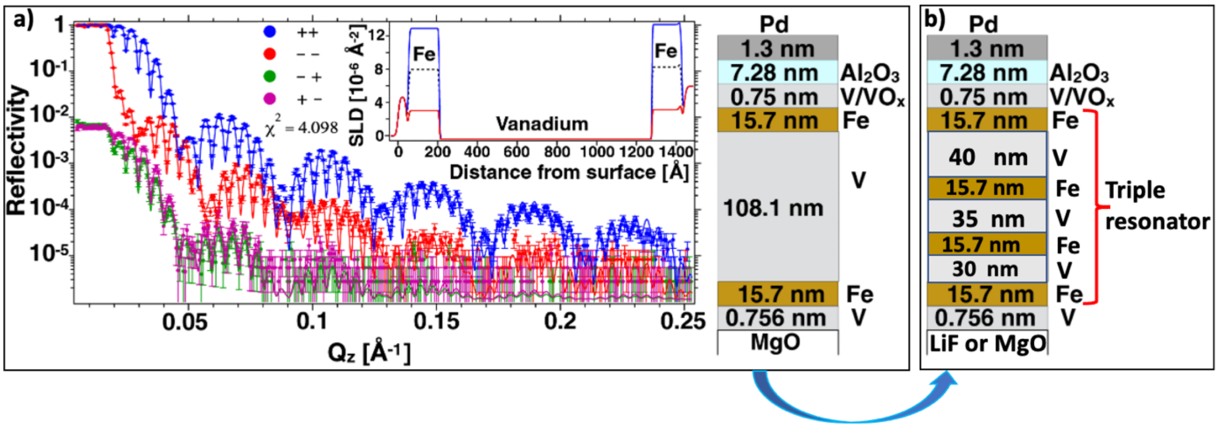

Figure 2. a) Neutron reflectivity and SLD profile (top right) of a Fe/V resonator deposited on a MgO substrate. Adapted from ref. [1]. b) A schematic of the new sample structure is shown on the right panel, two samples of triple resonator on LiF and MgO substrates.

4. Justification of beamtime

We will focus on the characterisation of the Fe/V triple resonator films, as we have done previously [1]. For the sample on MgO substrate, we will quantify the amount of gain in incoherent scattering at the superlattice in resonant condition, to estimate whether an inelastic experiment would be feasible at Super ADAM in the future. For the sample on LiF substrate, we will focus on the reflected signal, the background from substrate is low. We will compare the results from the two samples both in the incoherent detectors and PSD. Accounting for measuring the two multi-stack Fe/V samples, we kindly ask for 4 days of beam time on Super ADAM. If successful, our Fe/V triple resonator on LiF(001) substrate have the potential further improve the signal-to-noise ratio, via resonant-enhancement signal and absorb-suppress noise. A step forward to study dynamics in thin films and interfaces with neutron spectroscopy.

References

[1] M. Wolff, A. Devishvili, J. A. Dura, F. A. Adlmann, B. Kitchen, G. K. Pálsson, H. Palonen, B. B. Maranville, C. F. Majkrzak, and B. P. Toperverg, Phys. Rev. Lett. 123, 016101 (2019).
[2] Pan, Peng, Maxime Debiossac, and Philippe Roncin. "Polar inelastic profiles in fast-atom diffraction at surfaces." Physical Review B 104.16: 165415 (2021).
[3] Perrichon A, Devishvili A, Komander K, et al. Resonant enhancement of grazing incidence neutron scattering for the characterization of thin films[J]. Physical Review B, 103(23), (2021).
[4] Kuznetsov V, Lohstroh W, Rogalla D, et al. Neutron spectroscopy study of the diffusivity of hydrogen in MoS_2[J]. Physical Chemistry Chemical Physics, 23(13): 7961-7973, 2021.
[5] Xin, Xiao, et al. "Finite-size effects: hydrogen in Fe/V (001) superlattices." *Physical Review Letters* 113.4 (2014): 046103.
[6] Droulias S A, Pálsson G K, Palonen H, et al. Crystal perfection by strain engineering : The case of Fe/V (001)[J]. Thin Solid Films, 636: 608-614, (2017).